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QUALITATIVE ANALYSIS BY MEANS OF DROP-REACTIONS: A SYSTEMATIC STUDY OF INTERFERENCE

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In a previous paper (1), the authors presented a system of qualitative microanalysis by means of drop tests based on a sodium carbonate-sodium peroxide separation. As was pointed out at that time, the separation gives two groups, namely the "aqueous" group, which consists mainly of elements that yield water-soluble carbonates and hydroxides, and the "acid" group which is made up chiefly of elements that give insoluble carbonates and hydroxides. It was further pointed out that one of the main advantages of the system offered was that the oxidation by the sodium peroxide yields the elements in a constant state of oxidation, usually the highest. This adjustment of valence has two main advantages; first, since all forms of an element are converted to one common valence, many tests are eliminated; second, the elimination of extra valence forms reduces the possible interferences and makes interference studies much more simple.

Because so many tests reported in the literature are checked for only those interferences that might be found in the particular qualitative group in question, and because the type of interference found was seldom given, it was deemed advisable to check the tests used in the new system.

The reagents, apparatus and tests used were described in a previous paper (1). The solutions of "knowns" were made by fusing a weighed quantity of the substances to be investigated with sodium peroxide and sodium carbonate. The melt thus obtained was crushed, leached with hot water, and finally neutralized with nitric acid. Solutions obtained by the above procedure contained the elements in the same state of oxidation as would ordinarily be found in actual laboratory analysis using the new system.

METHOD OF INVESTIGATION

Because of the varied forms of phosphate interference, special attention was paid to the behavior of this element. The following outline of procedure was used throughout the investigation, and was applied in turn to each element considered.

1. Blank
2. Element under investigation
3. Element + phosphate
4. Periodic group I
   Periodic group II
   Periodic group III etc.
5. Periodic group I + element
   Periodic group II + element
   Periodic group III + element etc.
6. Periodic group I + phosphate
   Periodic group II + phosphate
   Periodic group III + phosphate etc.
7. Periodic group I + element + phosphate
   Periodic group II + element + phosphate
   Periodic group III + element + phosphate etc.

The interferences found were reported as being positive if a false test was obtained and negative if the test was prevented. Partial inhibiting of tests and masking of tests due to highly colored ions were also noted.

Elements investigated were:

Group I: Lithium, sodium, potassium, copper, silver, and gold.
Group II: Beryllium, magnesium, calcium, zinc, strontium, cadmium, and barium.
Group III: Boron and aluminum.
Group IV: Silicon, titanium, zirconium, tin, lead, and thorium.
Group V: Nitrogen, phosphorus, vanadium, bismuth, arsenic, and antimony.
Group VI: Sulfur, chromium, selenium, molybdenum, tungsten, uranium.
Group VII: Fluorine, chlorine, manganese, bromine, iodine.
Group VIII: Iron, nickel, cobalt.

CONCLUSIONS

A complete investigation of interferences has been made for the tests used in the detecting of thirty-nine elements. It was found that the interferences listed in the literature (2, 3, 4) were very incomplete in many cases, and in none of the tests described was the completeness of the interference studies given. Because of the lack of space a complete record of the present findings can not be included. However, the following typical example will serve to give an idea of the state of our present knowledge of interference.

ALUMINUM (Aqueous group)

1 d. soln. on a spot plate, 1 d. NaOH(N/10), 1 d. alizarin S., 2 d. acetic acid. In the presence of aluminum a pink color is obtained.

Run blank.

Interferences:

Blank.....Yellow color
Aluminum.....Pink color
Aluminum + HPO_4.....Pink color

Group	— Al	+ Al	— Al + HPO_4	+ Al + HPO_4
I	+ (1)			
II	+ (2)			
III	—	+	—	+
IV	+ (3)			
V	+ (4)			
VI	+ (5)			
VII	—	+	—	+
VIII	+ (6)			

1. Copper gave a positive interference. Large amounts of silver gave a slight off-color brownish tint to the blank and a deeper red to the aluminum color.
2. Barium gave a positive interference.
3. Zirconium, lead, and thorium gave positive interferences.
4. Vanadium gave a false test when present in large amounts. Very high concentrations of bismuth interfered positively.
5. Molybdenum and chromium interfered as they gave stains similar to that of aluminum.
6. Iron gave an off-color brown. Very large amounts of cobalt and nickel gave positive interferences.

Observations: The test drop should be alkaline prior to the addition of the alizarin sulfonate; the final acidity, however, should be below a paH of 4. Molybdenum, chromium and vanadium are the only interfering substances when the sodium carbonate-sodium peroxide separation is used. If they are found to be present they may be removed by precipitation with $BaCl_2$. The excess $BaCl_2$ must then be removed by adding $Ba(OH)_2$, evaporating to dryness, taking up in 2 d. H_2O and filtering.

The interferences listed for this test by Feigl are: Iron, cobalt, nickel, alkaline earths and chromates. Lange's Handbook gave iron, nickel, chromium and manganese as being interfering ions. As is shown by the above data, the interferences due to cobalt, manganese, calcium, and strontium could not be confirmed. In addition the following elements were found to give interferences although they were not listed by Feigl or Lange's Handbook: Copper, zirconium, lead, thorium, bismuth and molybdenum.

Of all the interferences found, only molybdenum, chromium, and vanadium appear in the same group as aluminum.

BIBLIOGRAPHY

1. SMITH, LOTHROP, and WEST, PHILIP. Proc. Iowa Acad. XLV.
2. FEIGL, FRITZ, "Spot Tests," Nordman Publishing Co., New York City, 1937.
3. *Lange's Handbook of Chemistry*, Handbook Publishers, Inc., Sandusky, Ohio, 1937.
4. BENEDETTI-PICHLER, A. A., "Introduction to the Microtechnique of Inorganic Qualitative Analysis," Microchemical Service, Douglaston, N. Y., 1935.

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